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Characterization of Carbon Fiber Emissions from Current and Projected Activities for the Manufacture and Disposal of Carbon Fiber Products

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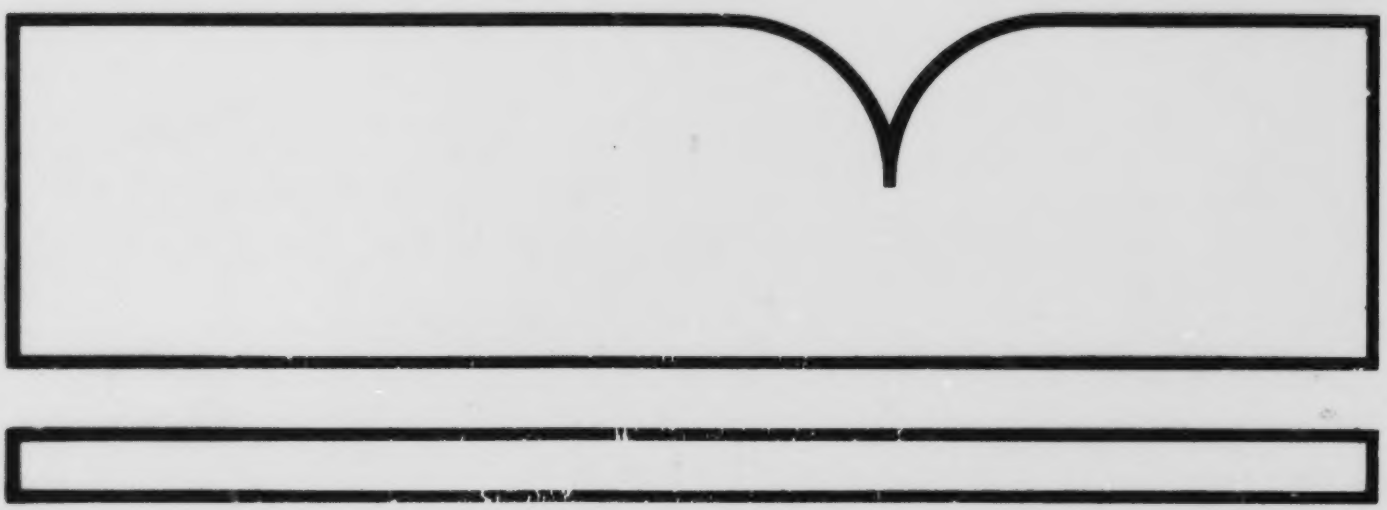

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CHARACTERIZATION OF CARBON FIBER EMISSIONS FROM CURRENT AND PROJECTED ACTIVITIES FOR THE MANUFACTURE AND DISPOSAL OF CARBON FIBER PRODUCTS

by

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16. ABSTRACT

Composite materials formed by impregnating a carbon or graphite fiber mat with plastic binders are being used increasingly in military, aerospace, sports and automotive applications. Carbon fibers are formed primarily from synthetic fibers carbonized in the absence of oxygen. Possibilities exist for the release of these fibers to ambient air during their formation, handling, weaving or impregnation, or during the manufacturing or incineration of composites. This study was concerned with characterizing the rate and physical/chemical properties of such emissions.

Samples were collected from manufacturing operations. Operations considered included fiber winding, prepregging and weaving, as well as composity cutting, grinding, drilling, machining, sanding, and incineration. Release rates (fiber mass released per unit of material processed) ranged over several orders of magnitude with the largest releases being associated with weaving and incineration. Except for incineration where fiber diameters were reduced somewhat by burning, the original fiber diameters were maintained in the emitted material. Fiber lengths varied over wide ranges from tens to thousands of micrometers. Incineration experiments suggested that mechanical agitation and air flow in the incinerator would strongly affect releases. Electrical characterization of the fibers demonstrated that fibers will move and form chains in electrical fields.

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ABSTRACT

Composite materials formed by impregnating a carbon or graphite fiber mat with plastic binders are being used increasingly in military, aerospace, sports and automotive applications. Carbon fibers are formed primarily from synthetic fibers carbonized in the absence of oxygen. Possibilities exist for the release of these fibers to ambient air during their formation, handling, weaving or impregnation, or during the manufacturing or incineration of composites. This study was concerned with characterizing the rate and physical/chemical properties of such emissions.

Samples were collected from air streams controlling release of fibers from manufacturing operations or in the vicinity of such operations when no air flow control existed. Operations considered included fiber winding, prepregging and weaving, as well as composity cutting, grinding, drilling, machining, sanding and incineration. Release rates in terms of fiber mass released per unit of material processed in the operation ranged over several orders of magnitude with the largest releases being associated with weaving and incineration. Except for incineration where fiber diameters were reduced somewhat by burning, the original fiber diameters were maintained in the emitted material. Fiber lengths varied over wide ranges from tens to thousands of micrometers. Incineration experiments suggested that mechanical agitation and air flow in the incinerator would strongly affect releases. Electrical characterization of the fibers demonstrated that fibers will move and form chains in electrical fields and that for low power electronic equipment, the intrustion of fibers could cause significant damage or disruption of normal operation.

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SECTION 1

INTRODUCTION

Carbon and graphitic fibers are chemically inert, are resistant to high temperatures and thermal shock, have good mechanical strength, are light weight, and are good electrical conductors. Because of these properties, composites formed from the fibers combined with a binder material (such as epoxy or various plastics) are being employed in an increasing number of aerospace, military, commercial and industrial applications. Carbon fibers are made by heating organic fibers (natural or synthetic) at high temperatures in the absence of oxygen. The long lengths of synthetic fibers make them adaptable for lay-up and woven products.

Whenever carbon fibers are produced, handled, woven or impregnated with a binder, the manufacturing operations provide some opportunity for the fibers to break off and to be released into the ambient air. In manufacturing operations with the composite materials such as cutting, sanding, or machining, opportunities again exist for fibers to be released. Finally, release of fibers can occur during the use of composites or more significantly during disposal of the products through incineration.

Because of their physical properties, carbon fibers released to the ambient air may pose special problems. Of particular concern is the potential for carbon fiber interference with or shorting out of electrical and electronic devices. Although much is known about the carbon fibers as used in manufacturing operations, the nature of the released fibers is not well established either in terms of release rates or characteristics of the released fibers. Manufacturers, processors, and end-product users are usually aware of problems associated with carbon fiber release and in most cases utilize good housekeeping practices and protective or removal techniques to eliminate excessive carbon fiber dissemination into work and ambient atmospheres. To provide a basis for improving control methods and to develop information related to determinations of typical release

rates and characteristics, the physical and chemical properties of emitted carbon and graphitic fibers were characterized experimentally. The experimental measurements included determinations of:

(1) Mass concentrations, number concentrations, and size distributions

(2) Chemical, optical, and morphological properties

(3) Electrical and physical properties that cause problems in electrical and electronic devices.

SECTION 2

CONCLUSIONS

Based on the sampling and analyses of carbon fibers, experimental evaluation of their electrical properties, and studies of their burning characteristics, the following conclusions have been reached:

1. Of the manufacturing operations or processes investigated, drilling and weaving release the most carbon fibers.

2. The amount of carbon fiber released and the distribution of fiber lengths are highly dependent upon the particular machining or manufacturing operation. Fiber diameter is not significantly affected by any operation except incineration.

3. Releases of carbon fibers during incineration of composites will be dependent on the degree of mechanical agitation of the burning material and on the air flow rate in the combustion region. A typical emission rate of carbon fibers from the burning zone in a municipal incinerator would likely be of the order of one percent of the composite being burned.

4. Composites made with epoxy binder materials are expected to give a greater fiber release during combustion than those made with phenolic binder materials.

5. The burning rate for carbon fiber composites is a sensitive function of temperature with the burning rate increasing rapidly with temperatures in the range from 500 to 700 C (1000 to 1250 F).

6. The resistance of graphitized carbon fibers ranges between about 1×10^3 and 25×10^3 ohms per centimeter of fiber length and fibers between 0.6 and 1.25 cm long will conduct currents of about 5 to 15 ma before burning with power inputs of 0.2 to 0.5 W.

7. Fibers orient in electrical fields and will form chains to bridge gaps that are longer than individual fibers.

8. Carbon fibers will move in the direction of increasing field strength in nonuniform electrical fields, and in uniform fields will move back and forth

between plate electrodes as the fibers are charged, attracted and discharged successively within the field and on contact with the electrodes.

9. The risk to electrical and electronic equipment increases as the airborne concentration and carbon fiber length increase, and increases as the distance between circuit elements decreases.

10. Only with sudden infusion of large numbers of fiber would there be any risk to most electrical systems operating at 110 V and greater than 1 W; however, electronic equipment operating at low power levels could easily be damaged or be made unreliable, perhaps insidiously, by only a single fiber critically located.

SECTION 3

CARBON FIBERS EMITTED DURING MANUFACTURING OPERATIONS

FIELD PROGRAM DESIGN

As part of this study a limited field program was conducted to obtain indications of the level of carbon fiber emissions generated by each of several key manufacturing and processing operations. The philosophy of this investigation was to collect data representative of the actual process emission without regard to control strategy or abatement procedures. The investigation consisted of on-site collection of fibers emitted during typical manufacturing operations followed by microscopic analysis of fiber count and size leading to subsequent concentration level and size distribution findings. Factors considered in designing the study included: type of fiber being processed, manufacturing operation, accessibility of sites and location/proximity of sites.

Based on discussions with manufacturers and researchers, it can be estimated that of the three generic carbon fiber types: PAN, rayon and pitch, currently about 75 percent of the useage is PAN with most of the rest being rayon. Over the next ten years the percent useage of PAN should remain the same or increase with rayon useage being reduced to a nearly negligible level and the pitch useage increasing to account for the difference. Furthermore, a full 70 percent or more of the applications of carbon fiber is related to the aircraft industry with this market share likely to increase in the near future. Given this information the field study concentrated solely on PAN fibers and the uses related to the aircraft industry and its suppliers.

Figure 1 shows the production steps and fiber emission points for composites made from a PAN precursor. The major operations include (1) Filament Manufacturing, (2) Stabilizing and Carbonizing, (3) Weaving, (4) Prepregging, (5) Composite Manufacturing (Machining) and (6) Handling Steps. Because of industry sensitivities we were not able to measure emissions associated with

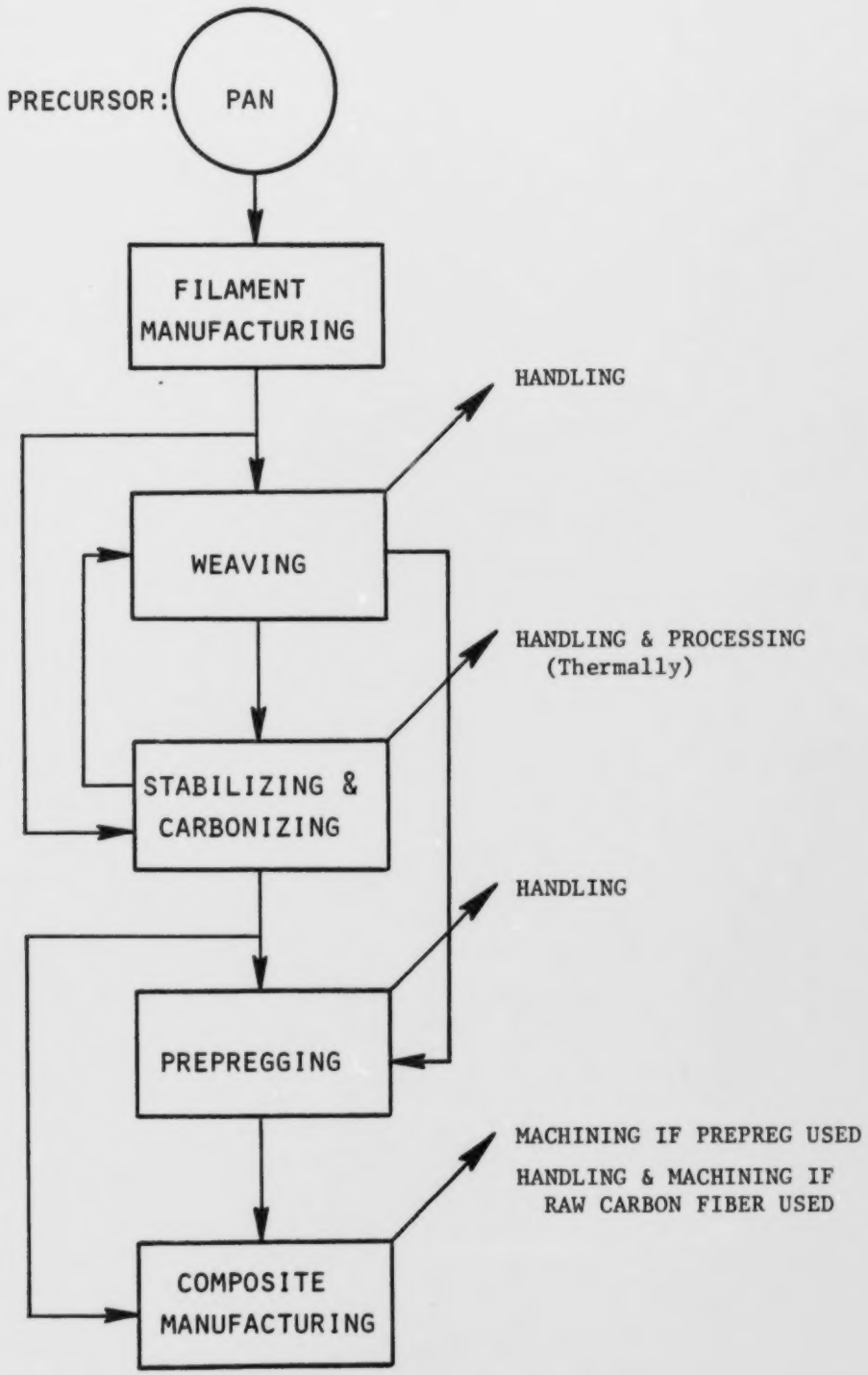


Figure 1. Production steps and fiber emission points for composites made from a PAN precursor.

filament manufacturing and stabilizing and carbonizing. The study did measure emissions of weaving, handling and prepregging the carbonized fibers and machining of the composite. Two types of weaving (rapier and shuttle) were investigated as well as a winding procedure and a "tape" type prepregging operation. The machining operations included cutting, drilling, routing and sanding.

SAMPLING LOCATION

The location of sampling was chosen for each operation to obtain a representative sample at all points of likely emission subject to the constraint of interfering with normal operations or worker activity. Table 1 presents a brief summary of the sample locations for the program. The type of sample configuration is also given (see following section). For situations where the emissions from an operation were not controlled or contained, an open-face type sampling arrangement was used. For situations where the emissions were contained in an exhaust duct, a sampling probe and in-line filter were used.

Some of the operations of concern (prepregging and weaving) were fairly large and open requiring samples at several points to obtain a proper characterization. Other operations (winding and most machining) were either enclosed or equipped with a vacuum system to control emissions. For these operations one sampling location often sufficed.

In addition to the locations indicated in Table 1 outside ambient samples were taken at each site.

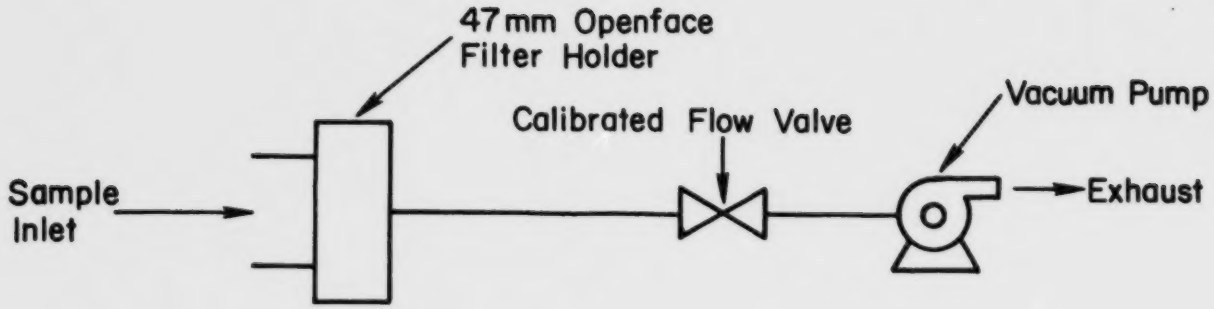
SAMPLING METHODS

The sampling procedure used in each circumstance was either an open-face type sample where no point source control was used (i.e., weaving, prepregging, some machining) or an in-line procedure in cases where the emissions were locally ducted (winding, most machining) to a control. In the latter case isokinetic sampling was conducted upstream of the control. Background samples were also taken with the open-face configuration. Figures 2A and 2B are schematics of the open-face and inline sample set-ups, respectively.

With both types of sampling configurations the procedure involved drawing the fiber laden air through high efficiency membrane filters with a vacuum pump. A 5.0 μm pore size, 47 mm diameter, cellulose ester membrane (Millipore) filter

TABLE 1. SAMPLING LOCATIONS

Operation	Type of Sample	Location
Prepregging	Open-Face	Above and Below Feed Spools Above and Below Guide Bar Above Tape Room Background
Weaving Shuttle Loom	Open-Face	Above and Below Feed Spools Above and Below Loom Feed Above and Below Harness Below Harness
Rapier Loom	Open-Face	Above and Below Feed Spools Above and Below Loom Feed Above Harness Above Rapier
Handling (Winding)	In-Line	At Three Locations in Vacuum Duct Leading from Enclosed Operation to Control Filter
Machining Edge Routing	In-Line Open-Face	In Vacuum Duct Used for Control 10 cm Above Routing Edge
Cutting		
Power Saw-Dry	Open-Face	10 cm Above Cut Surface
Power Saw-Wet	Open-Face	10 cm Above Cut Surface
Hand Held Knife	Open-Face	10 cm Above Cut Surface
Sanding	Open-Face	10 cm Above Sanded Surface During Sanding and Blowoff
Drilling		
Automatic	In-Line	In Vacuum Duct Used for Control
Hand-Held	In-Line	In Vacuum Duct Used for Control
Reaming	In-Line	In Vacuum Duct Used for Control



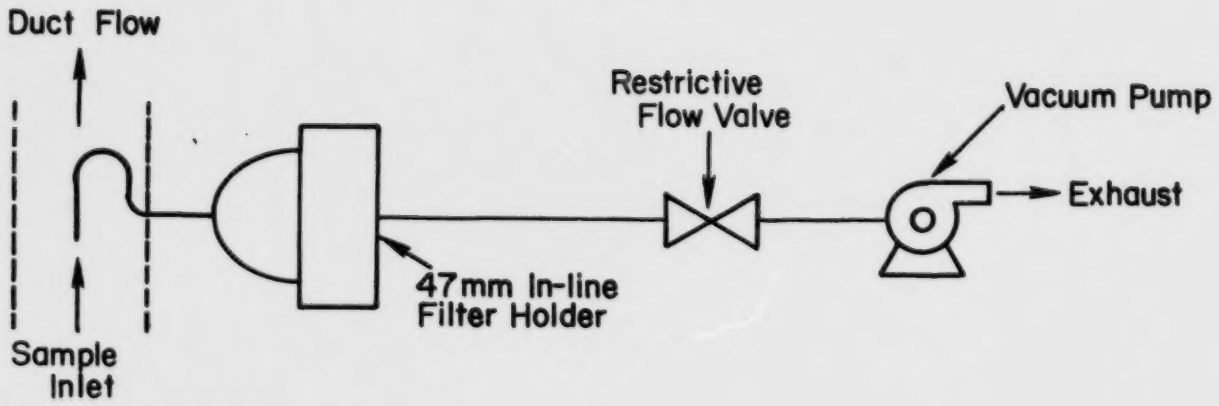


Figure 2. Sampling arrangements. (a) Open Face, (b) In Line

was the substrate used for fiber collection. Collections were made at representative locations for each process. The sampling flow rate was chosen for each case by considering time and anticipated concentration, and was then set using a calibrated restricting valve for the open-face configuration. For the in-line set-up the flow rate, again fixed by a restricting valve, was set at a level to provide, isokinetic sampling based on in-line flow measurements conducted immediately prior to sampling.

ANALYTICAL METHODS

Light Microscopy for Fiber Concentration and Size

Since carbon fiber sizes are large enough to be resolved by light microscopy, light microscopic analyses were performed on the collected samples. The specific analytical procedure for carbon fibers was designed to yield quantitative results which express numbers and mass of carbon fibers per cubic meter of sampled air and/or per unit mass of collected sample particulate.

Specimen preparation for light microscopy and the optical conditions employed during analysis were selected to eliminate interferences from extraneous organic particulate and from fibers other than carbon fibers. Carbon fibers being opaque produce high image contrast and therefore do not require phase contrast optics for detection. Consequently, glass fibers, cellulosic fibers, and transparent mineral fibers can be rendered almost invisible by mounting the filber-bearing dispersed particulate in a medium which has a refractive index near that of the transparent fibers and using a relatively large condenser aperture to observe the fibers in transmitted light. With this set up, only the opaque carbon fibers are visible.

The following sequential operations constitute the analytical procedure. (1) A sample collection integrated over the desired time period is made on a 47 mm diameter filter. (2) The entire collection on the filter is immersed in $\sim$10 ml of filtered deionized water and sonicated for 15 minutes. (3) 25 ml of concentrated nitric acid is added to the sonicated sample and the mixture is heated to 180 F to destroy the filter and extraneous organic particulate. (4) The suspension is diluted to 1000 ml and aliquots of 100, 200 and 700 ml filtered through 5.0 µm pore size, 47 µm diameter SM Millipore filters. (5) When the filters have dried, they are

transferred to 2 x 3 in. glass microscope slides where they are cleared with the NIOSH clearing solution (1:1 dimethyl phthalate and diethyl oxalate containing 0.5 g/ml of dissolved Millipore filter material) and covered with glass coverslips. (6) Counts and measurements of carbon fibers observed over measured filter areas are made and calculations of carbon fiber concentrations and size distribution are made from there data.

A wide range of magnifications is required to count and measure fibers which vary in length from a few micrometers to perhaps as great as 15 centimeters. The lower detection limit was about one micrometer and measurements at higher magnifications were made directly with a microscope equipped with an eyepiece micrometer which had been calibrated with a stage micrometer for the optical conditions employed during measurements. When photographic prints are used, for the lower magnifications, an enlargment of a photograph of a stage micrometer or other scale is made for calibration purposes. Sometimes both low and high magnification methods of measuring may be required on the same preparation due to a large range of fiber sizes. In this case, of course, the results from the two methods must be normalized on the basis of filter areas scanned and magnifications employed.

Fiber length and width measurements are used in the calculation of fiber volume by multiplying length times width ordiameter squared; volume in turn is converted to mass by multiplying by an assumed density of 1.85 g/cm^3. Concentrations are based on considerations of sampling times, air flow rates, filter areas, dilutions, aliquot sizes and magnifications.

Pregl-Dumas and EDS for Chemical Analysis

In addition to the concentration and size analysis, selected samples were subjected to chemical analysis for C, H and N and for overall elemental composition. The C, H and N measurements were conducted with a modified Pregl-Dumas technique. The elemental composition determinations were made for the elements including sodium and higher atomic weight elements with an Energy Dispersive Spectrometer (EDS) capability attached to the Scanning Transmission Electron Microscope (STEM).

RESULTS

Emission Level and Fiber Size

From the procedure noted above, the concentration and size of emitted fibers were determined. Table 2 shows a summary of the results for each of

TABLE 2. SUMMARY OF CARBON FIBER EMISSIONS

Operation	Average Fiber Length (μm)	Average Fiber Diameter (μm)	Fibers/m^3	Airborne Mass Concentration ng/m^3	Amount (a) Released
Handling (Winding)	2000	6.5	900	2.20×10^{5}	1.99×10^{-5} percent
Weaving					
Rapier Loom	1400	7.5	22,025	3.21×10^{6}	1.44×10^{-2} "
Shuttle Loom	1540	6.9	8,650	6.34×10^{5}	1.37×10^{-2} "
Prepregging	1800	5.7	6,450	6.98×10^{5}	2.62×10^{-4} "
Machining					
Cutting					
Power Saw (Dry)	40	5.5	1.45×10^{6}	3.22×10^{6}	5.25×10^{3} ng/cm
Power Saw (Wet)	25	7.8	3.96×10^{5}	1.03×10^{7}	7.84×10^{1} "
Hand Cut	580	6.9	3.24×10^{5}	1.40×10^{7}	7.69×10^{3} "
Routing	33	5.5	3.97×10^{7}	7.55×10^{7}	2.10×10^{3} "
Sanding	31	6.4	5.65×10^{5}	1.35×10^{6}	8.71×10^{2} ng/cm^2
Drilling					
Hand-Held	53	6.5	2.09×10^{7}	7.32×10^{7}	6.18×10^{6} ng/cm^3
Automatic	79	6.5	6.59×10^{8}	3.95×10^{9}	7.22×10^{6} "
Reaming	79	7.3	2.55×10^{6}	1.99×10^{7}	8.58×10^{6} "

(a) Amount released as percent of total mass of material handled in operation, mass per length of cut, mass per area sanded, or mass per volume of material removed in operation.

the processes investigated. The size and concentration numbers are weighted averages of several samples for most cases; however, a statistical basis was not used on obtaining or analyzing the data. Nevertheless the results are believed to be adequate for indicating the levels and characteristics of carbon fiber emissions. The handling, weaving, prepregging and wet cutting are all results from samples of the same fiber type. In all cases these samples showed concentration levels significantly lower than the process samples and thus did not influence the results or conclusions.

The size of the fibers measured is given in terms of mean length and mean diameter. Although the mean diameter was fairly consistent within a given sample and did not vary much from the diameter of the raw carbon fiber material, the lengths varied widely. The fiber lengths measured appear to be highly process dependent and varied greatly even within a given sample.

The last column in Table 2 is presented as an attempt to normalize the processes for comparative purposes. For the "line" processes a release percent of the total fiber processed is used. For the other processes the appropriate linear, areal or volumetric release amount is given. Following the discussions of the processes a scenario is given and evaluated to help quantitize the relative contribution of each operation to total carbon fiber emission.

Below is presented a brief discussion of the results obtained from each process studied.

Handling (Winding)--

Of all the handling processes the carbon fiber is likely to encounter, the high speed winding process (actually rewinding a fiber strand from one spool to another) appears to be most severe. As such it was included in the study. The results indicate that even in such a severe situation relative little emission occurs. Furthermore an emission from such a process is easily contained and would probably contribute little to any problems associated with carbon fiber release.

Weaving--

During the course of the study two generic types of looms were encountered: (1) rapier and (2) shuttle. Samples were taken for both types of loom during

weaving of the same type of carbon fiber. Table 3 shows the comparative results of this portion of the study giving data for key areas of possible fiber emission. The percent released is determined by dividing the total additive release (as calculated from collected samples) by the total amount processed for a given time period. A perusal of the data presented shows that the two types of looms produce similar results. The percent release total is relatively high when compared with other processes. In addition due to space and accessibility requirements weaving emissions are generally not well contained.

The amount of emission is a function of variable parameters for weaving. Two of these are rate of weaving and fiber type. The data given in Table 4 show the results of measurements taken at the same point for three different weave rates on similar shuttle looms. The variation over two orders of magnitude for concentration level show the strong dependence of emissions on just these two parameters.

Prepregging--

Two types of prepreg are prevalent for prepregging of carbon fiber: (1) tape and (2) resin. Both processes involve directing the fiber strands onto (into) the medium thus it was decided the one such operation would be appropriate for characterizing emissions. A tape type prepregging operation was selected. Samples were taken near fiber contact points. Table 5 shows the results of emissions from each of the key points for this process. Again the percent released is calculated by dividing the total amount of fiber released during the sampling period by the amount of material processed during that period.

Machining--

Several machining operations were investigated as part of the study. These consisted of either linear type (cutting and routing) operations on the material, areal type (sanding) operations or volumetric type (drilling and reaming) operations. This distinction is important in trying to normalize the emission amounts for comparative purposes. Normalizing on a percent basis was not practiced since the amount of emission is related to the degree of processing and not the amount of material being processed (e.g., drilling 10 holes in a 1 lb composite

TABLE 3. CARBON FIBER EMISSIONS FROM WEAVING OPERATIONS

Operation	Average Fiber Length (μm)	Average Fiber Diameter (μm)	Fibers/m^3	Airborne Mass Concentration (ng/m^3)	% Released
Shuttle Loom					
At feed spool	1642	5.5	5,009 Above 6,580 Below	6.65×10^{5} Above 4.01×10^{5} Below	2.21×10^{-4}
At loom feed	2342	7.7	1,317 Above 4,885 Below	1.48×10^{5} Above 1.31×10^{6} Below	2.74×10^{-3}
At harness and shuttle	1352	7.2	14,716 Above 19,000 Below	8.41×10^{5} Above 3.92×10^{6} Below	8.94×10^{-3}
				TOTAL	1.37×10^{-2}
Rapier Loom					
At loom feed	945	7.8	4,529 Above 14,837 Below	2.50×10^{5} Above 1.70×10^{6} Below	1.72×10^{-3}
At harness	1050	7.6	8,327 Above 15,837 Below	3.70×10^{5} Above 2.42×10^{6} Below	2.40×10^{-3}
At rapier	1525	7.8	54,285	9.42×10^{6}	8.18×10^{-3}
At feed spool	1692	5.5	9,333 Above 16,370 Below	5.77×10^{5} Above 1.85×10^{6} Below	2.09×10^{-3}
				TOTAL	1.44×10^{-2}

TABLE 4. COMPARISON OF CARBON FIBER EMISSIONS FROM DIFFERENT WEAVING RATES AND FIBER TYPES

	Average Fiber Length (μm)	Average Fiber Diameter (μm)	Fibers/m^3	Airborne Mass Concentration (ng/m^3)	% Released
Below Harness					
Shuttle Loom 1	1821	7.8	19,000	3.92×10^{6}	7.38×10^{-3}
Shuttle Loom 2	1250	7.1	12,563	1.45×10^{6}	3.47×10^{-3}
Shuttle Loom 3**	198	8.4	878	2.25×10^{4}	1.98×10^{-4}

*Tightly woven at slow rate.

TABLE 5. CARBON FIBER EMISSIONS FROM PREPREGGING OPERATION

Operation	Average Fiber Length (μm)	Average Fiber Diameter (μm)	Fibers/m^3	Airborne Mass Concentration (ng/m^3)	% Released
Prepregging					
At feed spool	1430	6.1	3,626 Below 3,620 Above	4.97×10^5 Below 2.20×10^5 Above	2.21×10^{-4}
At guide bar	2339	5.7	28,968 Below 8,932 Above	4.46×10^5 Below 1.32×10^6 Above	2.62×10^{-5}
Above tape	915	5.5	8,184	4.16×10^5	1.45×10^{-5}
				TOTAL	2.62×10^{-4}

is likely to produce much more emission than drilling 1 hole in a 10 lb composite). The figures listed in the last column of Table 2 reflect this normalization procedure.

As one might imagine, machining or abrasive destruction of material can create a great deal of emissions. However, unlike the situation with many of the other processes the emissions from machining operations are generally localized and thus easily controlled. In fact it was observed that local control was routinely employed and appeared to be generally effective.

Normalized Emission Level by Operation--

In order to evaluate the relative importance of each of the processes included, the following scenario is presented. Consider a 30 cm × 30 cm × 1/4 cm piece of woven PAN base composite. Such a composite would weigh approximately 320 g with 190 g (60 percent) being carbon fiber material. If we apply the previously presented data to such a piece we can estimate the total amount of carbon emission and the relative contribution of such from each process. Let us consider that the fiber is wound, woven and prepregged and that the resultant composite is subjected to a cross-cut (30 cm), total edge routing (120 cm), one side sanding (900 cm^2) and drilling of five 0.5 cm^3 holes. Table 6 shows the results of that analyses. Table 7 shows the detailed calculations.

From Table 6 it is apparent that the greatest potential for release comes from the weaving and drilling operations. Missing from the data base are the possibly significant releases from the manufacturing and carbonizing processes, and perhaps most significant, from incineration. Releases during incineration are discussed in a subsequent section of this report. Also it should again be pointed out that the data base was established and the assessment made without regard to control considerations.

Chemistry

Chemical analyses were conducted on selected fiber samples to determine the relative composition of carbon, hydrogen and nitrogen. In addition to the three types of PAN fibers investigated as part of the field study two different pitch base fibers and one rayon base fiber were analyzed. Table 8 shows the results of this analyses. As expected, carbon was overwhelmingly the most prominent constituent of the three in all cases.

TABLE 6. QUANTITATION OF RELATIVE RELEASE BY OPERATION

Operation	Amount Released	Mass Released (μg)	% of Total Release
	percent		
Manufacturing and Carbonizing	?	?	--
Handling	2.0×10^{-5}	38	0.1
Weaving	1.4×10^{-2}	2.7×10^{4}	57.7
Prepregging	2.6×10^{-4}	4.9×10^{2}	1.0
	ng/cm		
Cutting	6.5×10^{3}	2.0×10^{2}	0.4
Routing	2.1×10^{3}	2.5×10^{2}	0.5
	ng/cm^2		
Sanding	8.7×10^{2}	7.8×10^{2}	1.7
	ng/cm^3		
Drilling	7.3×10^{6}	1.8×10^{4}	38.6
	% Released		
Total	2.5×10^{-2}	4.7×10^{4}	100

TABLE 7. CALCULATIONS FOR QUANTITATION OF RELATIVE RELEASE BY OPERATION

Handling	$2.0 \times 10^{-5}\% \times 190\ g = 38\ \mu g$
Weaving	$1.4 \times 10^{-2}\% \times 190\ g = 2.7 \times 10^{4}\ \mu g$
Prepregging	$2.6 \times 10^{-4}\% \times 190\ g = 4.9 \times 10^{2}\ \mu g$
Cutting	$6.5 \times 10^{3}\ ng/cm \times 30\ cm = 2.0 \times 10^{2}\ \mu g$
Routing	$2.1 \times 10^{3}\ ng/cm \times 120\ cm = 2.5 \times 10^{2}\ \mu g$
Sanding	$8.7 \times 10^{2}\ ng/cm \times 900\ cm^{2} = 7.8 \times 10^{2}\ \mu g$
Drilling	$7.3 \times 10^{6}\ ng/cm^{3} \times 5\ holes \times 1/2\ cm^{3}/hole = 1.8 \times 10^{4}\ \mu g$
Total % Released	$4.7 \times 10^{4}\ \mu g/190\ g = 2.5 \times 10^{-2}\%$

TABLE 8. C, H AND N ANALYSIS

Operation	Fiber Type	% C	% H	% N
Weaving				
Shuttle Loom 1	PAN A	94.6	0.4	5.0
Shuttle Loom 2	PAN C	99.7	0.2	<0.1
Shuttle Loom 3	PAN B	99.8	0.2	<0.1
Rapier Loom	PAN A	92.6	0.4	7.0
Machining	PAN A	96.1	0.4	3.5
Winding	PAN C	99.7	0.2	<0.1
	Pitch A	99.7	0.2	<0.1
	Rayon	99.4	0.4	<0.1
	Pitch B	95.4	1.0	0.6

In addition to the C, H and N analyses elemental analyses were conducted in an attempt to learn more about the fibrous emissions from the processes. As part of the quality control the outside ambient sample taken with each test sample was also analyzed for elemental composition. All elements other than C, H and N identified in the test samples were also found in the ambient samples. Therefore, at the levels encountered, emissions other than carbon fibers were not detected as being significantly different than background.

SECTION 4

INCINERATION OF CARBON FIBER COMPOSITES

A process of potentially major significance for the release of carbon fibers to the environment is the incineration of manufacturing scrap materials or discarded final products made of carbon fiber composites. Since the polymeric binder materials in these composites burn at lower temperatures than do the carbon fibers, there is a potential for release of the fibers into the effluent gas flow from the incinerator. This effect, of course, is mitigated by the combustion of the fibers themselves. To evaluate the release of fibers from composite structures during combustion and to evaluate the effects of temperature and time on binder and fiber burning, several laboratory or small-scale and one larger scale incineration experiments were performed.

The general approach taken in the laboratory incineration experiments was to measure the weight loss as dependent on time and temperature for small pieces of composite materials. These experiments provided data on burning rates and fiber release from the composite materials. The larger scale experiment was performed as a joint effort between this study and a related study on "Development of a Method for Positive Identification and Measurement of Carbon Fibers Concentrations Emitted from Stationary Sources" (EPA Contract No. 68-02-3229). The results of the larger scale incineration experiments are being reported under the study noted above and will only be summarized here to provide an extrapolation of the small laboratory scale incineration experiments.

LABORATORY COMBUSTION EXPERIMENTS

The laboratory scale combustion experiments were intended to provide a basis for evaluating the rate of carbon fiber release during the incineration of composite materials. Composite materials with an epoxy binder and with a phenolic binder were studied. Weight loss as dependent on time and temperature were measured for each composite type. In addition to the burning rate data, observations of the physical changes in the composite materials provided information useful to an understanding of the composite combustion process.

Laboratory Combustion Apparatus and Procedures

The laboratory scale combustion experiments were conducted with the apparatus shown in Figure 3. The apparatur consisted simply of a vertical quartz glass tude, 3.2 cm (1 1/4 in.) in diameter, that was contained in a furnace and through which air was passed upward. Thermocouples positioned as noted in the figure were used to measure gas and furnace temperatures. A preweighed, small piece (about 1.2 x 1.2 x 0.6 cm) of composite material was placed in a stainless steel mesh basket and lowered into the tube to initiate the experiment. After a prescribed time the composite or the remaining residue was removed and weighed, and the physical condition of the remaining composite was noted. The entire air flow of 7.1 l/min (0.25 cfm) through the apparatus was sampled through a copper mesh screen onto glass fiber filters at the upper exit from the tube. The presence or absence of fibers on the screen and the filter was noted and the filter was weighed. The weight of material collected on the filter was consistently orders of magnitude less than the measured weight loss, and therefore has not been considered in this discussion.

Laboratory Combustion Results

In operating this combustion apparatus it was noted that at high 21 l/min (0.75 cfm), air flow rates, nearly all of the fibers released from the composite were carried upward and were deposited on the screen or filter. At lower flow rates nearly all of the fibers remained in the stainless steel basket. For this reason the lower air flow rate of 7.1 l/min (0.25 cfm) was used in the experiments since the burning of the fibers was being studied.

A second significant observation was that the fibers released from the composite had the same lengths as the dimensions of the composite piece. This indicates that the fibers were released intact without significant breaking along their lengths. The diameters appeared to be reduced and the fiber surfaces pitted by oxidation at higher temperatures or longer times.

The implications of these observations are that in an incinerator, the release rate and the lengths of fibers are dependent on air flow rates and mechanical agitation of disruptions in the incinerator. Further, the diameters of the emitted fibers will be dependent on burning rate at the incinerator conditions (temperature and oxygen levels), and on fiber residence time in the incinerator.

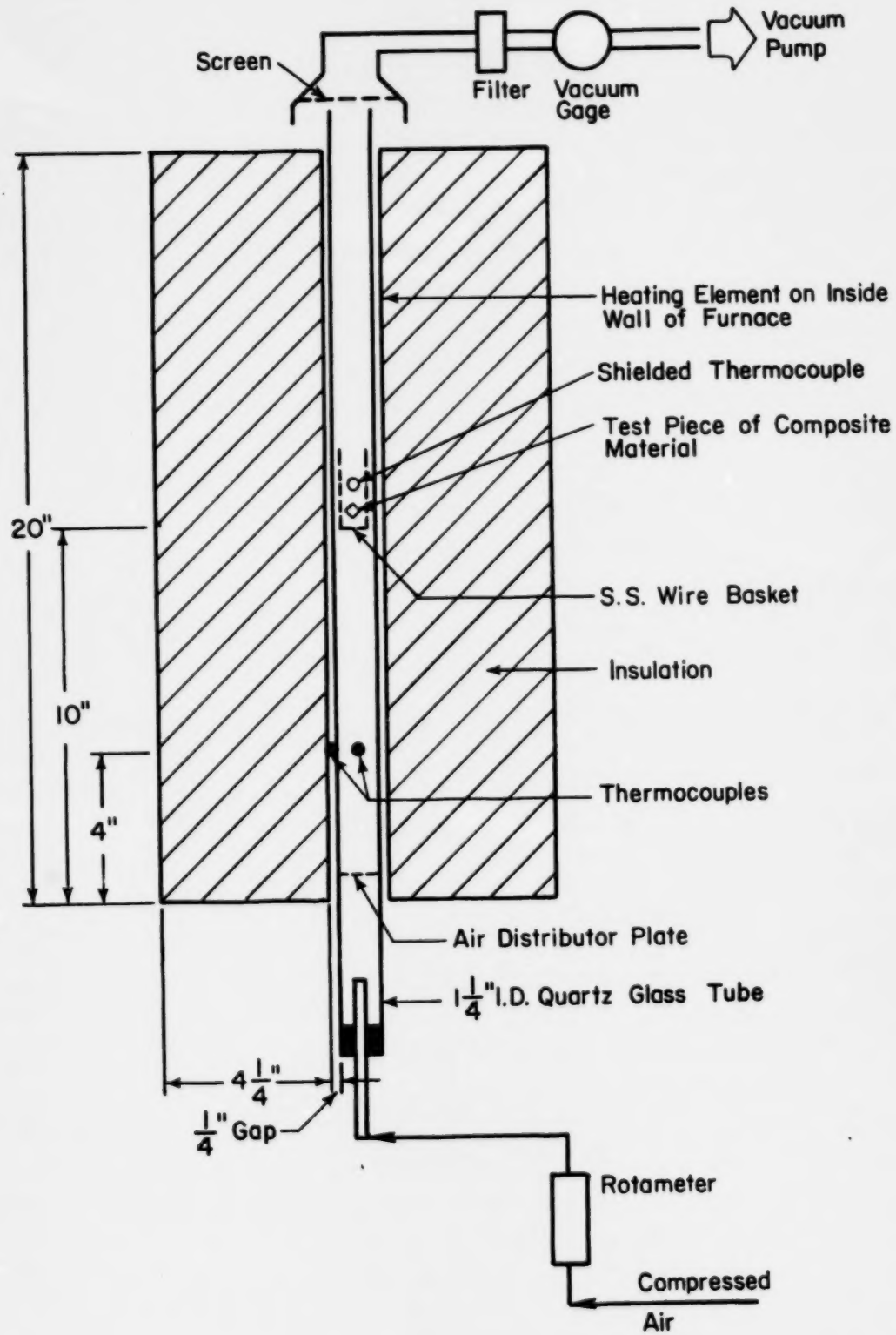


Figure 3. Small-scale laboratory incinerator apparatus.

The main thrust of these small scale experiments was to determine the combustion rate for the composite material given as percent mass loss as a function of different incineration times at selected temperatures. The results are presented in Figures 4 and 5 for composites formed with epoxy and phenolic resins, respectively. These data combined with observations of the samples provide an understanding of the differences between composites with epoxy and phenolic binders.

The effects of temperature on burning rate are shown in Figure 4 where the rates are seen to increase significantly with temperature. The epoxy binder was burned out of the samples first, thereby exposing or releasing the carbon fibers to the combustion air. After the epoxy was incinerated, the fibers themselves burned. The large gap in weight loss rates between 538 C (1000 F) and 677 C (1250 F) suggests that the carbon fibers have an ignition point between these temperatures. The composite materials were comprised of about 60 percent fibers and hence for all temperatures above the binder ignition temperature but below the carbon ignition temperature, the weight loss should approach 40 percent. Above the ignition temperature for the fibers, the weight loss should approach 100 percent at longer times.

For the phenolic binder, the behavior of the composite was different than that observed for the epoxy. The phenolic binger tended to swell, become porous, and leave a residue or char that burned at about the same rate as the carbon fibers. Hence, the fibers were not as readily released from the composite material for these experiments (note that there was no agitation of the sample). Rates of composite mass loss for the phenolic binder are seen to be lower than those for the epoxy binder case at the same temperatures. This difference apparently results from the manner in which the binders burn and expose the carbon fibers for combustion.

LARGE-SCALE LABORATORY INCINERATION

Carbon fiber composite materials were incinerated along with shredded and pelletized municipal refuse to determine the emission of carbon fibers from a process intended to model municipal incineration. As noted previously this was a joint effort with a related study on "Development of a Method for Positive Identification and Measurement of Carbon Fibers Concentrations Emitted from Stationary Sources" (EPA Contract No. 68-02-3229). Only highlights of the experiment and the major results are reported here. Experimental details are reported under the study noted above.

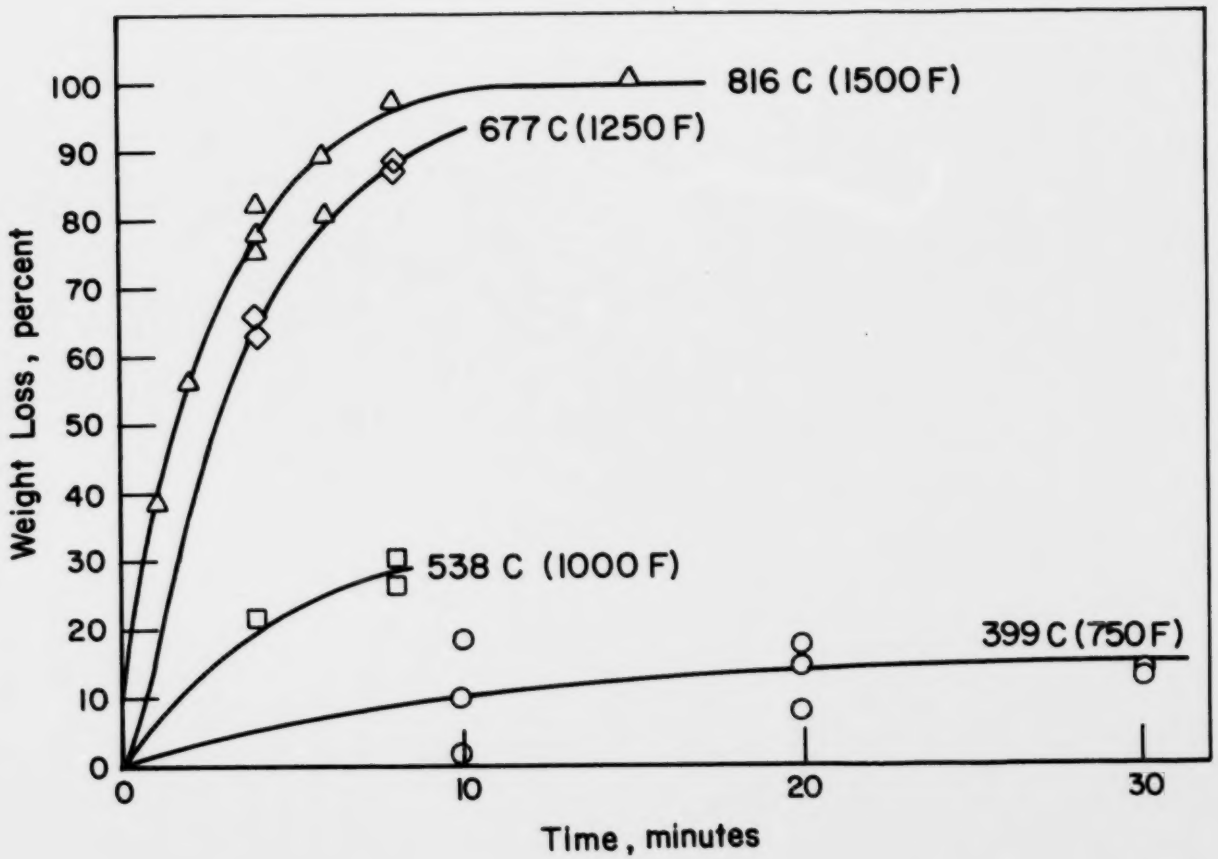


Figure 4. Incineration rates for composites with epoxy binder.

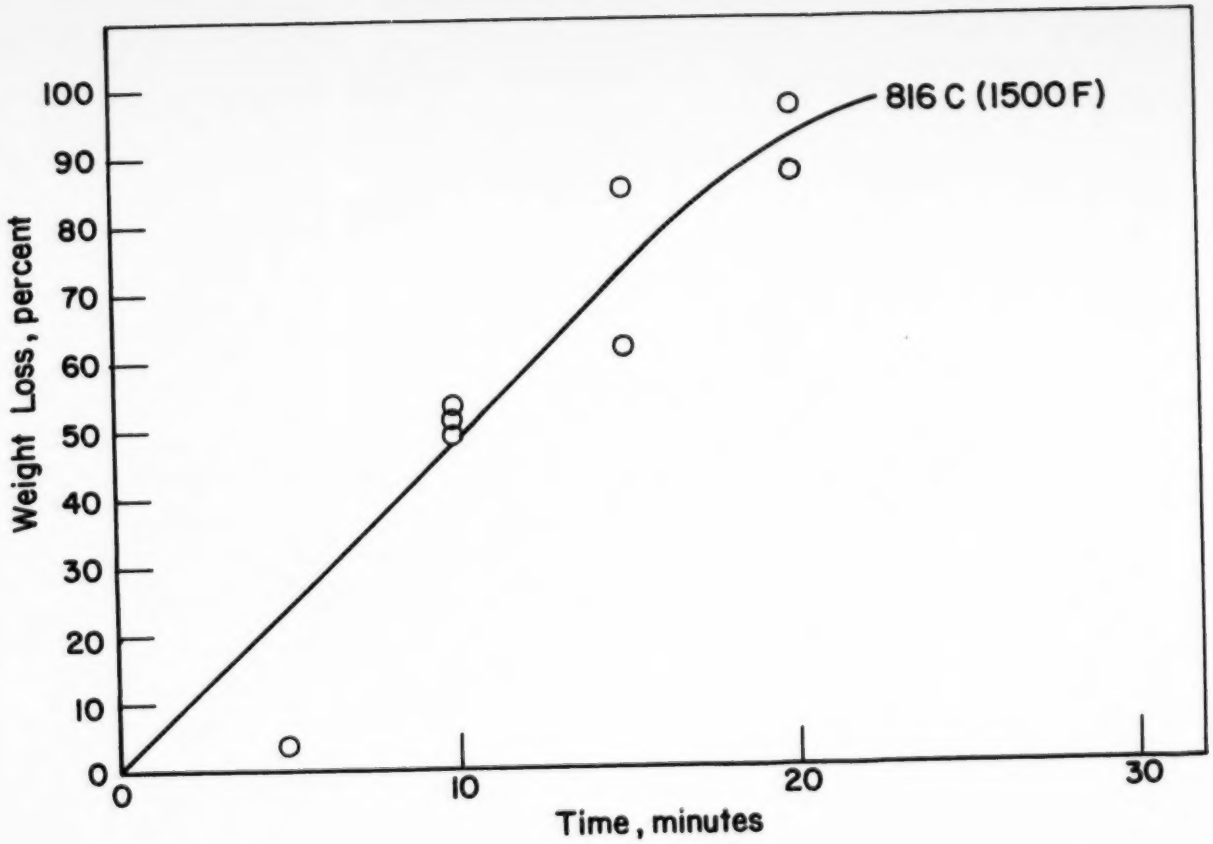


Figure 5. Incinerater rates for composites with phenolic binder.

Incineration Experimental Methods

The experiments were performed using a laboratory incinerator operating with refuse feed rates of 34 kg/hr (75 lb/hr). After steady state operation had been achieved, 450 g (1 lb) of the composite material was added to the feed over a one minute period. Sampling of effluent gases for particulate matter was performed using SASS and Method 17 procedures. In addition, samples were collected from inside the incinerator after the experiment. Weighings of probe and filter collections were supplemented by counting and sizing with an optical microscope to provide information on total mass and fiber emission rates and on fiber dimensions.

Incineration Results

Results of the larger scale incineration experiments showed that the concentration of fibers in the effluent gas were about 4000 fibers/m^3, the fiber diameters were reduced somewhat from original dimensions by oxidation in the incinerator, fiber length varied widely between about 50 and 5500 micrometers with nearly all of the fiber mass associated with lengths of about 1000 to 6000 micrometers, and the fraction of fibers released with the effluent gas stream being about one percent of that fed in the composite. About 90 percent of the total mass of fiber emissions was released within one hour of the time of composite introduction into the incinerator.

The overall results of the larger scale experiments are consistent with the results from the small scale incineration experiments and together they provide some understanding of the manner in which carbon fiber composites will burn and what can be expected for fiber emissions. Considering burning rates for the carbon fibers at usual incinerator temperatures, it can be concluded that the fibers will be released from the composite structure fairly rapidly, will be entrained in combustion gases at a rate dependent on both air flow rate and mechanical agitation in the incinerator, and will burn in the combustion zone to some extent dependent on temperature and oxygen concentration. Control of fiber emissions by removal from effluent gases should not be difficult since most fibers will be long enough for inertial devices to be effective.

SECTION 5

ELECTRICAL CHARACTERIZATION OF CARBON FIBER

Extensive studies were made to characterize the electrical properties of carbon or graphite fibers and to assess the potential hazard of such fibers to devices with electrical circuits. The electrical characterization included measurements of physical properties such as the resistance and current carrying capability of individual fibers and observations of fiber movements and interactions in electrical fields such as might exist in electrical equipment.

The fibers did not move or orient in plain magnetic fields; consequently, no work was done to characterize interactions with magnetic devices.

APPARATUS FOR MEASURING ELECTRICAL PROPERTIES OF FIBERS

Various techniques were considered for measuring the electrical resistivity of individual graphite fibers. A simple device was made with adjustable parallel electrodes that could be mounted on a microscope stage. Because of the fragile nature of the fibers, a clamping device could not be used and good electrical contact was made to the ends of the fibers with a drop of mercury.

The apparatus is shown schematically in Figure 6. Two slide blocks were mounted on two parallel 0.480-m (3/16-in.) diameter metal rods spaced 1.9 cm (3/4 in.) apart. A metal electrode with a Lucite insulator was fastened on each slide block. The desired fiber length was achieved by setting the gap between the slide blocks, positioning the fiber on the metal electrodes, and securing the ends with a drop of mercury. Suitable electrical connections were made for applying potentials to one electrode while the other electrode was grounded. Potentials were applied with a Heath Kit Model 1P-20, 0 to 50 V, d-c power supply* and the current through the fiber was measured with a Simpson Model-260 Multimeter†.

*Made by Heath Company, Benton Harbor, Michigan.
†Made by Simpson Electric Company, Chicago, Illinois.

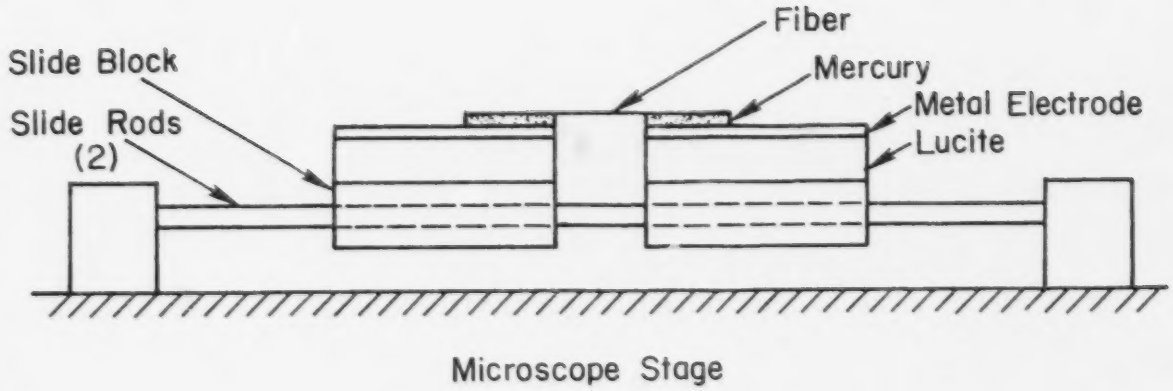


Figure 6. Apparatus for measuring resistance of fibers.

ELECTRICAL PROPERTIES OF CARBON FIBERS

Resistance measurements were made on 0.6 cm (1/4-in.) and 1.25 cm (1/2 in.) lengths of fibers as the applied potential difference across the ends of the fibers was increased from 1 volt to the burnoff voltage. The resistance, resistivity, burnoff voltage, and burnoff current for fibers of various sizes are summarized in Table 9.

Resistance (R) which is calculated from the applied potential difference (V) and measured current (I) is V/I. Resistance per unit length of fiber of length (L) is R/L. Resistivity is calculated from the relation of RA/L where A is the cross-sectional area ($\pi d^2/4$) of the fiber determined using the average diameter (d) of the fiber.

Thornel Fibers

The Thornel Graphite yarn (Grade WYG 130-1/2 PVA) was relatively uniform in diameter ranging from 6 to 8 μm. The electrical resistance per unit length of the Thornel yarn was relatively constant at 2.8×10^3 to 3.5×10^3 ohm-cm and the resistivity varied from about 8.8×10^{-4} to 1.8×10^{-3} ohms/cm. The resistance decreased as the voltage was increased to produce burnoff. The burnoff voltage, current, and power dissipation of the Thornel yarn were fairly reproducible. Fibers about 1.25 cm long burned off at an applied potential difference of 32 to 36 volts, and 0.6 cm long fibers burned off at 18 volts. The current through the fibers reached 14 to 15 ma before burnoff and the power dissipation was about 1/4 and 1/2 W for the 0.6 and 1.25 cm fibers, respectively, before burnoff.

Pitch Yarn

The graphite fiber made with the pitch precursor was highly variable in diameter ranging from about 8 to 38 μm. The resistance of the pitch yarn varied from about 2.8×10^2 to 2.6×10^3 ohm and the resistivity varied from about 1.2×10^{-3} to 2.4×10^{-3} ohms/cm.

The pitch fibers burned off at somewhat lower applied voltage differences and generally carried more current before burnoff because of their larger cross-sectional areas. Power dissipation in the pitch fiber before burnoff was only slightly more than in the Thornel yarn.

TABLE 9. ELECTRICAL PROPERTIES OF CARBON FIBERS

Fiber	Diameter, μm	Test Length, cm	Resistance at 1 volt, ohms/cm	Resistivity, ohm-cm	Pre-Burnoff Resistance, ohms/cm	Burnoff		
						Volts	ma	Watts
Thornel	6	0.6	2.8×10^3	7.9×10^{-4}	1.9×10^3	18	--	--
	6	1.25	3.1×10^3	8.8×10^{-4}	2.9×10^3	36	15	0.54
	7	0.6	3.1×10^3	1.2×10^{-3}	1.6×10^3	18	15	0.27
	7	1.25	3.1×10^3	1.2×10^{-3}	1.7×10^3	32	15	0.48
	8	0.6	3.1×10^3	1.6×10^{-3}	2.0×10^3	18	14	0.25
	8	1.25	3.5×10^3	1.8×10^{-3}	1.7×10^3	33	15	0.49
Pitch	20 to 24 (21)	1.25	6.0×10^2	2.1×10^{-3}	5.3×10^2	21	30	0.63
	19 to 38 (23)	0.6	2.8×10^2	1.2×10^{-3}	2.4×10^2	9	60	0.54
	19 to 38 (23)	1.25	3.5×10^2	1.5×10^{-3}	2.8×10^2	14	34	0.47
	18 to 28 (22)	1.25	4.9×10^2	1.9×10^{-3}	4.1×10^2	18	32	0.47
	22 to 28 (25)	0.6	4.9×10^3	2.4×10^{-3}	4.1×10^2	10	35	0.35
	10 to 14 (12)	0.6	1.4×10^3	1.6×10^{-3}	1.3×10^3	17	20	0.34
	8 to 11 (10)	1.25	2.6×10^3	2.0×10^{-3}	2.1×10^3	33	12	0.43
PAN	6.5	0.6	21.0×10^3	7.0×10^{-3}	7.9×10^3	33	6.6	0.22
		1.25	14.3×10^3	4.8×10^{-3}	6.9×10^3	60	6.9	0.41
	7.0	0.6	24.6×10^3	9.4×10^{-3}	8.4×10^3	30	5.7	0.17
		1.25	17.5×10^3	6.7×10^{-3}	7.4×10^3	60	6.4	0.38
	7.0	0.6	10.5×10^3	4.0×10^{-3}	6.5×10^3	25	6.2	0.15
		1.25	13.5×10^3	5.2×10^{-3}	5.7×10^3	55	7.6	0.42
	7.0	0.6	7.9×10^3	3.0×10^{-3}	5.8×10^3	32	8.6	0.27
		1.25	14.4×10^3	5.5×10^{-3}	5.5×10^3	58	8.3	0.48
	7.0	0.6	10.5×10^3	4.1×10^{-3}	7.6×10^3	34	7.1	0.24
		1.25	17.9×10^3	6.8×10^{-3}	10.0×10^3	65	5.4	0.35
	7.5	0.6	10.6×10^3	4.7×10^{-3}	6.5×10^3	35	8.6	0.30
		1.25	12.4×10^3	5.5×10^{-3}	6.3×10^3	55	6.9	0.38
	7.5	0.6	10.5×10^3	4.7×10^{-3}	7.7×10^3	35	7.6	0.27
		1.25	13.8×10^3	6.1×10^{-3}	7.0×10^3	55	6.2	0.34
	8.0	0.6	10.4×10^3	5.1×10^{-3}	6.8×10^3	29	6.7	0.19
		1.25	23.9×10^3	11.9×10^{-3}	6.6×10^3	60	7.2	0.43
	8.0	1.25	17.5×10^3	8.6×10^{-3}	6.4×10^3	56	6.9	0.39
	8.0	0.6	18.1×10^3	9.0×10^{-3}	8.8×10^3	30	5.6	0.16
		1.25	14.3×10^3	7.1×10^{-3}	8.0×10^3	55	5.4	0.30

PAN Fibers

The diameter of the PAN fibers varied from about 6.5 to 8.0 μm. The resistance of the fibers varied from about 7.9×10^3 to 24.6×10^3 ohms/cm at low applied voltage and decreased to the range of about 5.5×10^3 to 10.0×10^3 ohms/cm at burnoff. The resistivity of the fibers varied from about 3.0×10^{-3} to 11.9×10^{-3} ohm-cm under low electrical stress. The fibers 0.6 cm long burned off with applied potentials of about 29 to 35 V and power inputs of 0.17 to 0.30 W. Burnoff of the 1.25-cm fibers required about 55 to 65 V and about 0.30 to 0.48 W. Currents of about 5.4 to 8.6 ma were measured at burnoff. For comparison, current at burnoff with an ac potential difference of 48 V (rms) was 6.5 ma for a 0.6-cm fiber with an 8 μm diameter. The power was about 0.3 W with ac. Thus the electrical properties of the PAN fibers essentially were the same with either ac or dc as might be expected in a purely resistive fiber.

Incinerated Fibers

Two samples of incinerated fibers were examined including long fibers recovered from the bottom of the large-scale laboratory incinerator and short fibers collected in a cyclone at the exhaust from the incinerator.

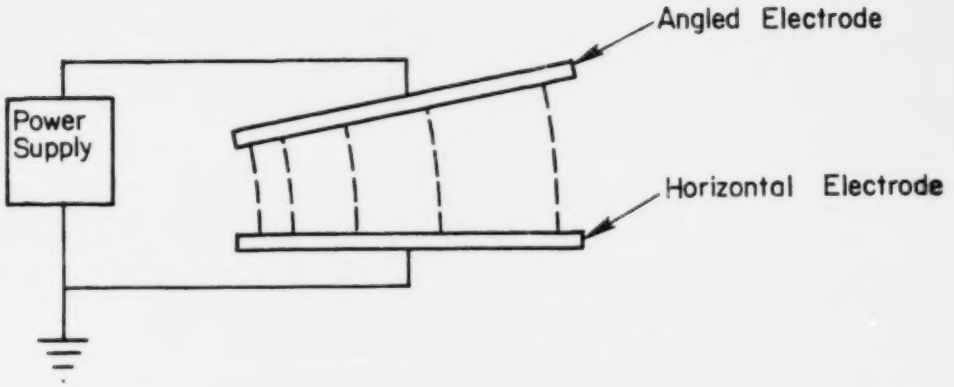
Table 10 summarizes the electrical properties of the incinerated fibers. Comparison of these measurements with those made on the original PAN fibers indicate that incineration generally decreased the resistivity and burnoff voltage, but increased the current at burnoff. As might be expected, the fibers collected in the cyclone showed the largest changes. Overall, the power required to burn off the bottom fibers was about the same as for the original fibers, but the power required to burn off the fibers collected in the cyclone decreased slightly.

FIBER MOVEMENT IN ELECTRICAL FIELDS

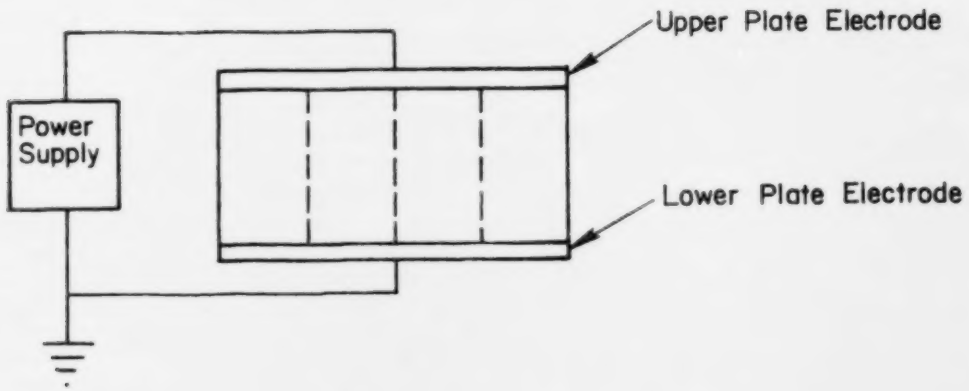
Movement of PAN and Thornel fibers was studied in uniform and non-uniform electrical fields with apparatus shown in Figure 7. The effects of field conditions on start of movement, rate of movement, and chaining of fibers were studied in various gaps with fibers from 0.3 cm (1/8 in.) to 2.54 cm (1 in.) long.

TABLE 10. ELECTRICAL PROPERTIES OF INCINERATED CARBON FIBER

Material	Diameter, μm	Test Length, cm	Resistance at Test, ohms/	Resistivity, ohm-cm	Pre-burnoff Resistance, ohms/cm	Burnoff		
						Volts	ma	Watts
Bottom Fiber	7	0.6	$4.2\ 10^{3}$	$1.6\ 10^{-3}$	$4.8\ 10^{3}$	25	8.6	0.21
Bottom Fiber	7	0.6	$5.5\ 10^{3}$	$2.0\ 10^{-3}$	$6.2\ 10^{3}$	22	6.0	0.13
Bottom Fiber	8	0.6	$5.5\ 10^{3}$	$2.7\ 10^{-3}$	$3.8\ 10^{3}$	25	11.0	0.27
Cyclone Fiber	6.5	0.4	$6.3\ 10^{3}$	$2.1\ 10^{-3}$	$6.3\ 10^{3}$	17	6.8	0.11
Cyclone Fiber	6.5	0.5	$8.0\ 10^{3}$	$2.6\ 10^{-3}$	$5.4\ 10^{3}$	20	7.4	0.15
Cyclone Fiber	7.0	0.4	$4.2\ 10^{3}$	$1.6\ 10^{-3}$	$4.0\ 10^{3}$	14	9.0	0.12
Cyclone Fiber	7.0	0.3	$6.0\ 10^{3}$	$2.3\ 10^{-3}$	$5.3\ 10^{3}$	13	8.2	0.11



(a)



(b)

Figure 7. Apparatus for producing (a) nonuniform electrical fields and (b) uniform electrical fields.

A nonuniform field was produced by applying a potential difference* between two 14-cm (5 1/2-in.) by 30-cm (12-in.) metal plates spaced 1.2 cm (1/2 in.) apart at one end of the long axis and 5.1 to 8.0 cm (2 to 3 1/2 in.) apart at the other end, with the lower plate in a horizontal position. In this configuration as shown in Figure 7 (a), the field lines are segments of circles with centers at the apex of the projected plates. The field is strongest at the end with the narrow gap and decreases as the gap between the plates increases.

A uniform field was produced by applying a potential difference between two parallel 15-cm (6-in.) square metal plates spaced 5.1 (2 in.) apart in a horizontal position as in Figure 7 (b). The electrodes were enclosed with Lucite sheets and Mylar films to prevent fiber drift from air drafts. The fiber movement in the nonuniform field was observed in a darkened room with a 75-W lamp to illuminate the field area along the axis of the plates. The strobe light for illumination.

A 100-megohm resistor in series with the power supply prevented burning the fibers when single fibers or chains of fibers shorted out the field electrode.

Movement in Nonuniform Field

The fibers were introduced into the nonuniform field in the region near the end of the plates where the gap was the smallest (1.25 cm)

Data in Table 11 show that fibers of all lengths from 0.3 to 1.25 cm (1/8 in. to 1/2 in.) moved from a position in the high field region to a position where the field strength is weaker in the nonuniform electrical field. The fibers acquired a charge by conduction while in contact with either electrode when the field strength was sufficiently high to overcome gravity and/or surface attractive forces. On contacting the second electrode, the charge reversed and the fibers jumped back and forth in the field. Centrifugal force produced as the fibers moved on the curved field lines exceeded the dielectrophoretic force of the nonuniform field and caused the fibers to move away from the apex of the plates. The fibers eventually reached a region where the field strength was either insufficient to lift the fibers or to impart

*Voltages were applied with a 0-15 kv dc Model AD3-315 power supply made by Wabash Magnetics, Inc., Huntington, Indiana.

TABLE 11. MOVEMENT OF PAN FIBERS IN NONUNIFORM FIELD

Fiber Length, cm	Fiber Length, inch	Plate Angle, degrees	Potential Difference, volts	Spacing At Final Position, cm	Spacing At Final Position, inch
0.3	1/8	7	500	2.5	1
			600	2.9	1-1/8
			800	3.2	1-1/4
			900	4.4	1-3/4
			1000	>5.1	>2
0.6	1/4	7	500	2.5	1
			600	2.9	1-1/8
			700	3.8	1-1/2
			800	4.8	1-7/8
1.2	1/2	7	500	>5	>2
0.3	1/8	12	500	4.4	1-3/4
			1000	6.3	2-1/2
0.6	1/4	12	500	6.1	2-3/8
			1000	7.0	2-3/4
0.9	3/8	12	500	6.6	2-5/8
			1000	7.6	3
1.25	1/2	12	500	7.0	2-3/4*
			1000	8.4	>3-1/2*

* The long fibers reached the Mylar film at the end of the electrode.

enough velocity to produce the centrifugal force required to cause further movement away from the apex of the plates. The fibers remained at this point. The gap between the plates at the final fiber position, as shown in Table 11, increased with fiber length and applied potential difference.

Movement in Uniform Fields

To characterize movement of the fibers in uniform electrical fields, individual fibers of various lengths were placed in a prone position on the lower of two parallel flat electrodes, and an increasing potential difference was applied between the electrodes until each fiber was lifted off the electrode. The two horizontally-oriented 15 cm^2 (6 $in.^2$) field electrodes were enclosed with sheets of Lucite and Mylar film to block drafts which cause the fibers to drift out of the field between the electrodes.

Thornel Fiber--

Data for Thornel fibers are summarized in Table 12. Each data point is the average of measurements on ten fibers. As might be expected higher applied potentials and stronger fields were required to lift shorter fibers in a given gap. Also as the plate spacing was increased to 3.8 cm only about 50 percent of the 0.35 cm fibers could be lifted with the maximum potential of 21 kv that was applied on the electrodes.

PAN Fiber--

The effect of fiber length and gap were studied in more detail with PAN fibers. Fibers were chopped to lengths of 0.15 cm (1/16 in.), 0.31 cm (1/8 in.), 0.62 cm (1/4 in.), 1.25 cm (1/2 in.), and 2.54 cm (1 in.) and each fiber was observed under various field conditions in gaps up to 4 times the fiber length.

Single fibers and groups of fibers were placed on the lower electrode and the electrode was tapped to induce movement of the fiber.

0.15-cm Fiber--In studies of the 0.15-cm (1/16 in.) PAN fiber, the gap between the plates was varied in steps from 0.32 cm (1/8 in.) to 0.48 cm (3/16 in.) and 0.64 cm (1/4 in.). The potential difference between the plates was increased in steps from about 40 to 70 V to about 400 to 800 V depending on the gap. The results are summarized in Table 13. At low voltages, the fibers oriented in the field. As the voltage was increased, the fibers began to jump from the lower electrode to the upper electrode when the electrode was tapped.

TABLE 12. FIELD REQUIRED TO LIFT THORNEL FIBERS

Plate Spacing, cm	Average Applied Field, kv/cm		
	0.35-cm Fiber	0.64-cm Fiber	1.25-cm Fiber
1.2	2.9	3.1	0.95
1.9	2.2	2.5	1.0
2.5	5.2	4.2	1.2
3.2	4.4	1.7	1.9
3.8	3.0	2.7	1.4
4.5	2.3	1.7	1.6
5.1	2.1	1.9	1.2

TABLE 13. MOVEMENT OF 0.15-cm PAN FIBER

Observed Effect	Applied Potential Range, volts		
	0.32-cm gap	0.48-cm gap	0.64-cm gap
Fiber orients in field	<40	60	70
Fiber jumps when tapped	40 to 50	70	80
Fiber continues jumping for 1 to 15 seconds after tapping	60 to 300	80 to 300	90 to 400
Fiber jumps continuously	>400	>400	>500
Fibers form chains	70	100	No chains formed

TABLE 14. MOVEMENT OF 0.32-cm PAN FIBER

Observed Effect	Applied Potential Range, volts		
	0.63-cm gap	0.95-cm gap	1.27-cm gap
Fiber orients in field	--	50	100 to 150
Fiber jumps when tapped	50	75	200
Fiber jumps from 1 to 2 seconds after tapping	50 to 150	100 to 200	200 to 900
Fiber jumps continuously	200 to 1100	300 to 1100	1100
Fibers form chains	75 to 400	150 to 500	150 to 400

At first, single jumps occurred; however, as the voltage was increased, the fibers jumped back and forth between the electrodes and the jumping continued for increasing periods after the tapping was stopped. Finally, at potential differences greater than 400 to 500 V the fibers jumped continuously. As might be expected, somewhat higher voltages were required to produce the observed effects in the larger gaps.

0.32 cm Fiber--With 0.32-cm (1/8-in.) PAN fiber, the gap between the electrodes was varied in steps from 0.63 cm (0.25 in.) to 0.95 cm (0.375 in.) and 1.27 cm (0.5 in.) while the applied potential difference was increased from about 75 V to 1,100 V. The results are summarized in Table 14. At low voltages, the fibers oriented in the field. As the voltage was increased, the fibers began to jump from the lower electrode to the upper electrode when the electrode was tapped. At first single jumps occurred; however, as the voltage was increased, the back-and-forth jumping continued for increasing periods after the tapping was stopped. Finally, the fibers jumped continuously. Somewhat higher voltages were required to produce the observed effects in the larger gaps.

0.64 cm Fiber--With 0.64-cm (1/4-in.) PAN fiber, the gap between the plates was varied in steps from 1.25 cm (1/2 in.) to 1.88 cm (3/4 in.) and 2.54 cm (1 in.), and the applied potential difference was increased in steps from about 75 to 150 V to about 400 to 1100 V depending on the gap. The results are given in Table 15.

2.54 cm Fiber--The movement of 2.54-cm (1-in.) PAN fiber was observed in 5 cm (2 in.), 7.5 cm (3 in.), and 10 cm (4 in.) gaps with applied potential differences of 200 to 7000 V. The long fibers in the large gaps acted distinctly different from the shorter fibers. In the 5 cm gap, the 2.54 cm fiber jumped back and forth between the plates with increasing frequency as the potential difference was increased from about 250 V to 1500 V. However, the fibers did not jump unless the plate was tapped. In the 7.5 cm and 10 cm gap, the fibers jumped between the plates with potential differences of about 300 to 2000 and 400 to 3000 V, respectively, when they were inserted into the field without first contacting the electrode. Potential differences of 2500 and 4000 V caused the fibers to jump when the electrode was tapped after the field was applied. In the 10 cm gap, the jumping became erratic with periods of floating as the potential difference was increased to 6000 V. At 7000 V, the fiber oscillated near the lower electrode without bouncing to the upper electrode. These observations are summarized in Table 16.

TABLE 15. MOVEMENT OF 0.64-cm PAN FIBER

Observed Effect	Applied Potential Range, volts		
	1.25-cm Gap	1.88-cm Gap	2.54-cm Gap
Fiber orients in field	75	125	150
Fiber jumps when tapped	75 to 100	125 to 150	175
Fiber continues jumping for 1 to 10 seconds after tapping	100 to 200	150 to 250	200 to 400
Fiber jumps continuously	250 to 700	300 to 1100	500 to 1100
Fibers form chains	100 to 400	150 to 400	400

TABLE 16. MOVEMENT OF 2.54-cm PAN FIBER

Observed Effect	Applied Potential Difference, volts		
	5-cm gap	7.5-cm gap	10-cm gap
Fiber orients in field	<200	<350	200 to 350
Fiber jumps when introduced into field	200	270 to 2000	400 to 3000
Fiber jumps continuously	250 to 1500	>2500	>4000
Fibers form chains	200 to 3000	-	-

Rate of Jumping and Movement--

Table 17 provides data concerning the potential difference required to lift the PAN fibers of various lengths in a uniform field with a plate spacing of 5 cm (2 in.). Lower potentials were required to lift longer fibers because the fiber itself reduced the gap and increased the effective strength of the applied field. Strobing the fibers showed that increasing the applied potential difference increased the fiber velocity as reflected by the increasing number of jumps per minute (N) made by the fibers. The fiber velocity can be calculated by multiplying N by the plate gap. At an applied potential difference of 1200 V, the 1.2-cm (1/2 in.) fibers had an average velocity of about 4050 cm/min (1620 in./min). Table 18 provides data about the number of jumps per minute made by individual 1.25-cm (1/2-in.) PAN fibers in different gaps as a function of the potential difference applied to the electrodes. The gap between the plates was varied in steps from about 1.6 cm (5/8 in.) to 3.7 cm (1 1/2 in.). The applied potential difference was varied up to 1000 V in the small gaps and up to 2500 V in the largest gap.

In the 1.6-cm gap, the jumping rate was too fast to measure accurately with a strobe light at potential differences greater than 1000 V. However, in the 3.7-cm gap, the jumping rate reached a maximum at an applied potential difference of about 2500 V and decreased slightly as the potential difference was increased to 3000 V. Higher voltages caused the fibers to move erratically and rapidly to the sidewalls.

Chaining of Fibers in Nonuniform Fields

Various studies were made to define conditions that produce chaining in electrical fields independent of wall effects. Nonuniform electrical fields were produced with pairs of concave and convex curved surfaces such as shown in Figure 8. Electrode spacing and applied potential difference were varied.

The electrodes were designed to produce nonuniform fields that can be used to concentrate the fibers in a specific field area as the fibers bounce back and forth between the electrodes. The convex electrodes were about 10 cm (4 in.) in diameter with a curvature of 12-cm (4 3/4 in.) radius. The concave electrodes were 9 cm (3 1/2 in.) in diameter with a radius of curvature of about 12.5 cm (5 in.). The electrodes were positioned with the curved surfaces in a diametric orientation such that the convex electrodes produced the highest

TABLE 17. JUMPING RATE OF PAN FIBERS IN 5-cm GAP IN UNIFORM FIELD

Fiber Length, cm	Fiber Length, in.	Starting Potential, volts	Jumps Per Minute (N) 400 volts	500 volts	600 volts	700 volts	800 volts	900 volts	1000 volts	1100 volts	1200 volts
0.3	1/8	530	-	-	-	41	84	115	122	155	177
0.6	1/4	440	-	-	93	144	177	205	257	300	335
0.9	3/8	390	-	100	137	220	263	375	485	535	617
1.2	1/2	350	82	175	255	310	370	467	560	690	810

TABLE 18. JUMPING RATE OF 1.25-cm PAN FIBERS IN UNIFORM FIELD

Applied Potential Difference, volts	1.6-cm Gap	2.2-cm Gap	2.8-cm Gap	3.7-cm Gap
200	2,300	600	-	-
300	2,470	1,280	300	160
400	2,610	2,080	750	310
500	2,730	3,220	1,080	415
600	3,590	3,970	1,480	550
800	11,200	6,200	2,500	960
1,000	-	8,900	3,500	1,380
1,200	-	-	-	2,000
1,400	-	-	-	2,580
1,600	-	-	-	3,360
1,800	-	-	-	3,940
2,000	-	-	-	4,150
2,500	-	-	-	4,250

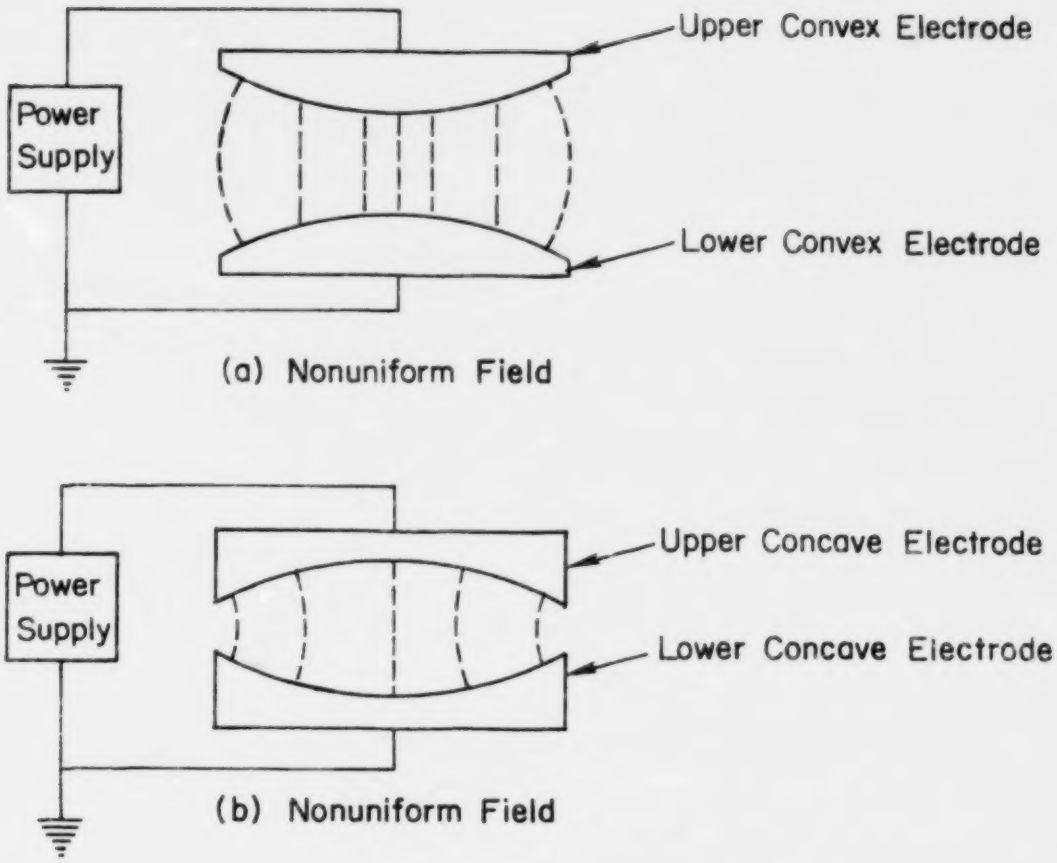


Figure 8. Electrodes for producing nonuniform electrical fields.

field intensity at a line between the centers of curvature of the opposing surfaces. The concave electrodes produced the lowest field intensity at this position. Dielectric forces on polarized fibers should move the fibers to the area of the strongest field while the usual field force on charged fibers should move the fibers to the weakest field area.

With a 3.2-cm (1 1/4 in.) gap at the center of the concave electrodes, an applied potential of 900 V or greater caused 1.2-cm (1/2-in.) PAN fibers to migrate toward the centerline of the electrodes. However, no chaining was observed even when relatively large numbers of fibers were placed in the gap. When the applied potential difference was about 900 V or lower, the charge-field forces were relatively weak and dielectrophoretic forces caused the fibers to drift toward the high field areas at the outer edge of the electrodes. In the outer edge gap which was about 1.75 cm (11/16 in.), the 1.2-cm (1.2 in.) fibers frequently formed chains and burned off. To avoid burning off the fiber chains, a 100-megohm resistor was placed in series with the power supply to limit the current. In this way, when fiber chains were formed the applied voltage dropped and movement of other fibers in the gap ceased.

Subsequently, the convex electrodes were set up to form the highest field intensity at the center of the electrodes, and fibers were inserted at the center area. However, when the field intensity was sufficient to cause the fibers to bounce, centrifugal force causes the fibers to move away immediately from the center area into weaker field areas and no chaining occurred.

Chaining of Fibers in Uniform Fields

The best chaining studies were performed in a uniform electrical field between a pair of 15 cm (6 in.) square plates. The electrodes were enclosed with sheets of Lucite and Mylar to block drafts that might cause the fibers to drift out of the gap between the electrodes. A 100-megohm resistor was placed in series with the electrodes to prevent burnoff whenever the filters bridged the gap. Initially, only two 1.25-cm (1.2 in.) fibers were placed in the electrical field at each plate spacing. Chains unexpectedly formed more readily in gaps that were much larger than the fiber length and in the weaker electrical fields. In a 1.6-cm gap, no chains were formed in two fiber systems. The jumping rates were greater than 2,300 per minute in all cases; and when the fibers came within about 1.25 to 2.0 cm (1/2 to 3/4 in.) of each other, they

repelled one another. In a 2.2-cm gap, the two fibers formed chains in a few seconds. The chained fibers generally did not bridge the gap and the fiber pairs continued to jump between the electrodes. The joined fibers frequently separated and rejoined. The rate of forming the chain increased with the jumping rate. At potential differences under 400 V, the fibers formed chains quickly when they were within 1.25 cm (1/2 in.) of one another and the chains did not break up. The chained fibers often stopped moving at 200 to 400 V. In the 2.8-cm (1 1/8 in.) gap, two fibers chained and separated rapidly at 1000 V. At 800 V down to 400 V, the chains remained together progressively longer: and at 300 V, the chained fibers were relatively stable. In a 3.7-cm (1 1/2 in.) gap, chains formed when the fibers were within about 2.5 cm (1 in.) of one another at 2000 V. This separation between fibers at which chaining occurred rapidly decreased with decreasing voltage and was only about 0.9 cm (3/8 in.) at 300 V.

When small groups of ten fibers and larger groups of 20 fibers were introduced into the gaps, chaining occurred more rapidly as might be expected. The larger numbers of fibers did not produce chains that bridged the gaps at 2.2, 2.8, and 3.7 cm. The groups of 10 fibers did not form chains at 800 to 1000 V but did form chains in 10 sec or less at 600 V and lower. Groups of 20 fibers chained at all voltages generally in 1 sec or less. In the 2.8 cm and 3.7 cm gaps, fibers both in the small groups and in the larger groups formed chains between the electrodes at all voltages. At 600 to 800 V, some short chains formed and jumped back and forth without bridging the gap. In all cases, the bridging occurred more rapidly with the larger group of fibers as might be expected. Chaining also occurred most frequently when the fibers were introduced as a bundle rather than as a number of individual fibers. Other observations of chaining with 0.5-cm, 0.32-cm, 0.64-cm and 2.54-cm PAN fibers are included in Tables 12, 13, 14 and 15, respectively.

The 0.15-cm (1/16 in.) fibers formed chains only in the two narrow gaps which were two and three fiber lengths. No chains were observed in the 4 X gap. Chains began to form at 70 to 100 V in areas where fiber concentration was high and increased with increasing voltage. However, at voltages which produced continuous jumping, chain formation stopped. The fibers spread apart rapidly as they jumped between the electrodes.

The 0.32-cm (1/8 in.) fibers formed chains in 2 X, 3 X and 4 X gaps.

The 0.62 cm (1/4 in.) fibers formed chains readily only in the gaps which were two and three fiber lengths. Chains were observed in the 4 X gap only at 400 V. Chains began to form at 100 to 150 V in areas where fiber concentration was high and increased with increasing voltage. However, at voltages which produced continuous jumping, chain formation gradually stopped as voltage was increased. The fibers spread apart rapidly as they jumped between the electrodes.

Chaining of 2.54-cm (1-in.) PAN fibers occurred readily in the 5 cm gap at potential differences as low as 300 V with separated fibers and 200 V with clusters of fibers. Five fibers generally formed chains that shorted the electrodes in 2 sec or less at applied potential differences of 1000 to 3000 V. Chaining in the 7.5-cm and 10-cm gaps was not investigated because the fibers moved rapidly to the walls of the field unit.